

2

REPORT DOCUMENTATION PAGE

Form Approved
CMB No. 0704-0188**AD-A232 368**

1b. RESTRICTIVE MARKINGS

3. DISTRIBUTION/AVAILABILITY OF REPORT

Approval for public release; distribution unlimited

5. MONITORING ORGANIZATION REPORT NUMBER(S)

4. PERFORMING ORGANIZATION REPORT NUMBER
Technical Report No. DU/DC/TR-186a. NAME OF PERFORMING ORGANIZATION
Department of Chemistry
Duke University6b. OFFICE SYMBOL
(If applicable)7a. NAME OF MONITORING ORGANIZATION
Office of Naval Research6c. ADDRESS (City, State, and ZIP Code)
Durham, NC 277067b. ADDRESS (City, State, and ZIP Code)
300 North Quincy Street
Arlington, VA 22217-50008a. NAME OF FUNDING/SPONSORING ORGANIZATION
Office of Naval Research8b. OFFICE SYMBOL
(If applicable)9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
N00014-89-J-1545; R&T Code 41350088c. ADDRESS (City, State, and ZIP Code)
800 North Quincy Street
Arlington, VA 22217-5000

10. SOURCE OF FUNDING NUMBERS

PROGRAM
ELEMENT NO.
NRPROJECT
NO.
053TASK
NO.
841WORK UNIT
ACCESSION NO.11. TITLE (Include Security Classification)
See Attached Title12. PERSONAL AUTHOR(S)
R.L. Wells, L.J. Jones, A.T. McPhail, and A. Alvanipour13a. TYPE OF REPORT
Technical13b. TIME COVERED
FROM _____ TO _____14. DATE OF REPORT (Year, Month, Day)
1991-02-2515. PAGE COUNT
2116. SUPPLEMENTARY NOTATION
Accepted for publication in the Journal of Organometallic Chemistry

17. COSATI CODES

FIELD	GROUP	SUB-GROUP

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
indium-arsenic, synthesis, structure

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

See Attached Abstract

DTIC
ELECTE
MAR 11 1991
S B D

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT

☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS21. ABSTRACT SECURITY CLASSIFICATION
Unclassified22a. NAME OF RESPONSIBLE INDIVIDUAL
Richard L. Wells22b. TELEPHONE (Include Area Code)
(919) 684-6404

22c. OFFICE SYMBOL

OFFICE OF NAVAL RESEARCH

Grant NOOO14-89-J-1545

R&T Code 4135008

Technical Report No. DU/DC/TR-18

SYNTHESIS AND CHARACTERIZATION OF INDIUM-ARSENIC COMPOUNDS
CONTAINING A FOUR-MEMBERED $\overline{\text{In-As-In-As}}$ OR $\overline{\text{In-As-In-Cl}}$ RING: CRYSTAL
STRUCTURES OF $[(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2]_2$ and
 $(\text{Me}_3\text{SiCH}_2)_2\overline{\text{InAs}(\text{SiMe}_3)_2\text{In}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}}$

by

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Prepared for Publication in Organometallics

Duke University
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February 25, 1991

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**Synthesis and Characterization of Indium-Arsenic
Compounds Containing a
Four-Membered $\overline{\text{In-As-In-As}}$ or $\overline{\text{In-As-In-Cl}}$ Ring:
Crystal Structures of $[(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2]_2$
and $(\text{Me}_3\text{SiCH}_2)_2\overline{\text{InAs}(\text{SiMe}_3)_2\text{In}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}}$**

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(Received January 11, 1990)

The first four-membered ring compounds with bridging of the indium atoms by two arsenic atoms and with mixed bridging of the indium centers by one arsenic and one chlorine, $[(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2]_2$ (1) and $(\text{Me}_3\text{SiCH}_2)_2\overline{\text{InAs}(\text{SiMe}_3)_2\text{In}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}}$ (2), to be characterized completely were prepared by the reaction of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ and $(\text{Me}_3\text{Si})_3\text{As}$ in a 1:1 and 2:1 mole ratio,

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respectively. X-ray crystallographic analyses showed both the $\overline{\text{In-As-In-As}}$ ring of **1** and the $\overline{\text{In-As-In-Cl}}$ ring of **2** to be planar. Compound **1** belongs to the orthorhombic system, space group $Pbcn$ (D^{14}_{2h}), with four molecules in a unit cell of dimensions: $a = 12.920(1)$, $b = 21.618(2)$, $c = 18.797(2)$ Å. Compound **2** crystallizes in the monoclinic system, space group $C2/c$ (C^6_{2h}), with four molecules in a unit cell of dimensions: $a = 19.233(3)$, $b = 10.798(2)$, $c = 20.819(3)$ Å, $\beta = 105.91(1)^\circ$. Compound **1** reacts with two equivalents of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ to yield **2** as a major product, as evidenced by ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR and melting point data.

Introduction

Recently, organogallium-arsenic compounds containing four-membered rings consisting of mixed-bridging of the gallium atoms by one arsenic atom and one halogen atom have been synthesized *via* dehalosilylation between an organogallium halide and a silylarsine.¹⁻⁴ Thus, compounds formulated as $\text{R}_2\overline{\text{GaAs}(\text{SiMe}_3)_2\text{Ga}(\text{R})_2\text{Cl}}$ ($\text{R} = \text{Ph}^{1,2}$, $\text{Me}_3\text{SiCH}_2^3$) and $\text{Ph}_2\overline{\text{GaCl}(\text{Ph})_2\text{Ga}(\text{Me}_3\text{Si})\text{As}(\text{CH}_2)_3\text{As}(\text{SiMe}_3)\text{Ga}(\text{Ph})_2\text{ClGaPh}_2^4$ have been described by us. Until now, our studies regarding the formation of such mixed bridge systems involving the heavier elements of Groups III and V have focused only on gallium and arsenic. In order to investigate the generality of this behavior, analogous reactions utilizing an organoindium halide were carried out. Here we report the isolation of $[(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2]_2$ (**1**) from the reaction of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ and $(\text{Me}_3\text{Si})_3\text{As}$ in a 1:1 mole ratio, whereas the reaction in a 2:1 mole ratio afforded $(\text{Me}_3\text{SiCH}_2)_2\overline{\text{InAs}(\text{SiMe}_3)_2\text{In}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}}$ (**2**). In addition to

describing the synthesis and complete characterization of **1** and **2**, including their crystal structures, the conversion of **1** to **2** is reported.

Experimental Section

General Considerations. All manipulations were performed using Schlenk techniques in an I²R glove bag under dry nitrogen or in a Vacuum/Atmospheres HE-43 Dri-Lab containing an argon atmosphere. Aromatic solvents were distilled from sodium/benzophenone ketyl under dry nitrogen. Benzene-d₆ was dried over sodium/benzophenone ketyl and vacuum transferred from the same. Pentane was distilled over LiAlH₄ under dry nitrogen. Indium(III) chloride was purchased from Alfa Products, Ward Hill, MA and was not further purified. (Me₃SiCH₂)₃In and (Me₃SiCH₂)₂InCl,⁵ and (Me₃Si)₃As⁶ were prepared by literature procedures. ¹H and ¹³C NMR spectra were obtained on a Varian XL-300 (300 and 75.4 MHz, respectively) spectrometer using 5 mm tubes. All spectra were referenced to TMS using the residual protons or carbons of deuterated benzene at δ 7.15 ppm or δ 128 ppm. NMR tubes were flame sealed under vacuum. All melting points (uncorrected) were obtained with a Thomas-Hoover Uni-melt apparatus and capillaries were flame sealed under argon. Crystals used in the x-ray analyses were flame sealed in 0.7 mm thin-walled glass capillaries. Elemental analyses were performed by E+R Microanalytical Laboratory, Inc., Corona, New York.

Preparation of [(Me₃SiCH₂)₂InAs(SiMe₃)₂]₂ (1**).** (Me₃SiCH₂)₂InCl (0.500 g, 1.54 mmol) in 30 mL of benzene and (Me₃Si)₃As (0.454 g, 1.54 mmol) in 5 mL of benzene were combined in a 100-mL one-necked round-bottomed flask equipped with a Teflon valve and a micro-stirbar. The solution, which was freeze-pump-thawed once and left to stir

at room temperature, became light yellow within one hour. Continuation of the stirring at room temperature for 3 days, followed by removal of volatiles *in vacuo*, afforded a yellow solid. The crude product was dissolved in a small amount of warm pentane and, after one day at $-15\text{ }^{\circ}\text{C}$, the solution afforded colorless crystals of **1** suitable for single-crystal X-ray analysis. Removal of pentane by evaporation afforded a white powder which, when redissolved in hot pentane followed by cooling to $-15\text{ }^{\circ}\text{C}$, gave a second crop of **1** (0.578 g total, 75.5% yield) [mp not observed (sample decomposed to a black solid above $210\text{ }^{\circ}\text{C}$)]. Anal. Calcd (Found) for $\text{C}_{28}\text{H}_{80}\text{As}_2\text{In}_2\text{Si}_8$: C, 32.94 (32.95); H, 7.90 (7.91); Cl, 0.00 (0.31). ^1H NMR: δ 0.24 (s, 8 H, CH_2), 0.32 (s, 36 H, SiMe_3), 0.49 (s, 36 H, SiMe_3). ^{13}C $\{^1\text{H}\}$ NMR: δ 3.60 (SiMe_3), 3.91 (CH_2), 5.32 (SiMe_3).

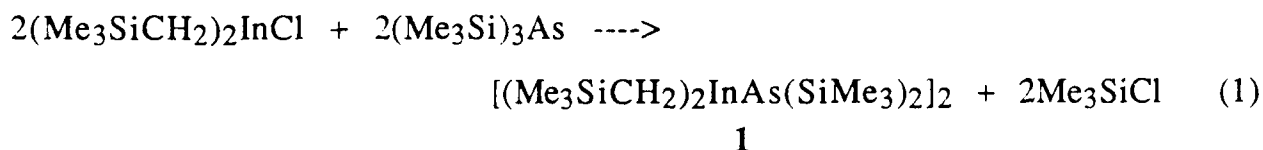
Preparation of $(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2\text{In}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}$ (2**).** $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ (0.500 g, 1.54 mmol) in 30 mL of benzene and $(\text{Me}_3\text{Si})_3\text{As}$ (0.227 g, 0.771 mmol) in 5 mL of benzene were combined in a one-necked round-bottomed flask equipped with a Teflon valve and a micro-stirbar. The solution was freeze-pump-thawed and, on stirring at room temperature, became yellow within an hour. After stirring the reaction mixture at room temperature for 3 days, the volatiles were removed *in vacuo*. Dissolution of the resulting solid yellow residue in pentane, followed by cooling to $-15\text{ }^{\circ}\text{C}$ for 5 days, afforded colorless crystals of **2** suitable for single-crystal X-ray analysis (0.103 g, 16.2% yield), m.p. $68\text{--}71\text{ }^{\circ}\text{C}$. Anal. Calcd (Found) for $\text{C}_{22}\text{H}_{62}\text{AsClIn}_2\text{Si}_6$: C, 31.64 (31.39); H, 7.48 (7.39); Cl, 4.24 (4.57). ^1H NMR: δ 0.27 (s, 44 H, CH_2SiMe_3), 0.39 (s, 18 H, SiMe_3); an individual methylene resonance was not observed. ^{13}C $\{^1\text{H}\}$ NMR: δ 2.83 (SiMe_3), 7.75 (CH_2), 4.69 (SiMe_3).

Reaction of 1 with $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$. $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ (13.1 mg, 0.040 mmol) and **1** (20.4 mg, 0.020 mmol) were combined in an NMR tube into which was vacuum distilled 0.75 mL of benzene- d_6 . After freezing the contents of the tube with liquid nitrogen, the tube was flame-sealed and allowed to warm to RT. After thawing, the ^1H NMR spectrum of the solution contained three singlets at δ 0.17, 0.26 and 0.39, the first of which corresponded to the SiMe_3 protons of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ (the methylene proton resonance at δ 0.27 is under the peak at δ 0.26). No peaks attributable to **1** were observed, even though the NMR tube still contained solid which was presumed to be **1**. After 1.5 hours, all of the solid in the tube had disappeared, and the ^1H and ^{13}C $\{^1\text{H}\}$ NMR spectra of the resulting solution corresponded to those obtained for **2**. After opening the tube, the solvent was removed and a colorless solid was isolated, the melting point of which was the same as that recorded for an authentic sample of **2**.

Structural Analyses of 1 and 2. Crystallographic data and measurements are presented in Table 1. Intensity data were corrected for the usual Lorentz and polarization effects; empirical absorption corrections (and in the case of **1**, a linear decay correction) were also applied. For **1**, non-hydrogen atom coordinates of its isomorphous gallium analogue³ were used as initial values, whereas the crystal structure of **2** was solved by direct methods. Positional and thermal parameters (at first isotropic, then anisotropic) for both compounds were adjusted by means of several rounds of full-matrix least-squares calculations. Hydrogen atoms were included at their calculated positions during the final least-squares iterations which also included refinement of an extinction correction. For structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from reference 7.

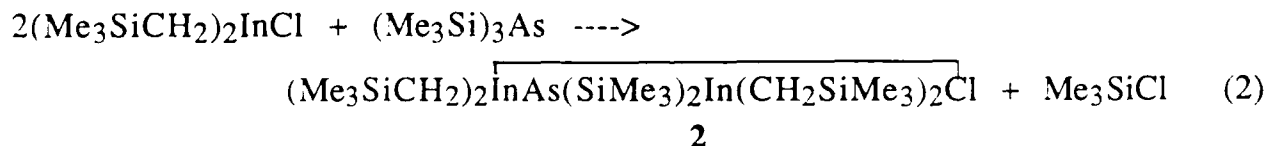
Results and Discussion

The reaction of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ and $(\text{Me}_2\text{Si})_3\text{As}$ in a 1:1 mole ratio at room temperature in benzene yields **1** (eq 1), the indium analogue of $[(\text{Me}_3\text{SiCH}_2)_2\text{GaAs}(\text{SiMe}_3)_2]_2$ (**3**).³ The ^1H NMR spectrum of **1** is consistent



with expectations based on its solid-state structure. Compound **1** is stable at room temperature over a period of at least 10 days, as evidenced by ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectroscopy.

When $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ is treated with $(\text{Me}_3\text{Si})_3\text{As}$ in a 2:1 mole ratio at room temperature in benzene compound **2**, the indium analogue of $(\text{Me}_3\text{SiCH}_2)_2\text{GaAs}(\text{SiMe}_3)_2\text{Ga}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}$ (**4**),³ is produced (eq 2). The ^1H



NMR spectrum of **2** shows only one type of trimethylsilyl and trimethylsilylmethyl group, with the methylene signal of the latter degenerate with its methyl signal. The $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum contains three signals which can be assigned to methylene or methyl carbons based on intensities. Compound **2** is unstable at room temperature, decomposing to an orange/brown solid within a few hours.

Dehalosilylation between an organogallium halide and a silylarsine to form gallium-arsenic bonds now has ample precedent^{1-4,8,9}; however, analogous reactions utilizing organoindium halides to form indium-arsenic bonds have until now been unknown. The synthesis of **1** and **2** is clearly dependent on the stoichiometry of organoindium halide and silylarsine. Thus, whereas compound **1** is prepared in good yield from the reaction of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ and $(\text{Me}_3\text{Si})_3\text{As}$ in a 1:1 mole ratio, **2** is formed in significantly lower yield when a 2:1 mole ratio is employed. The low yield of the latter is, in part, due to its high solubility in all solvents from which recrystallization was attempted (pentane, toluene, ligroin and benzene). This dependence on reagent stoichiometry to prepare **1** and **2** is interesting since it contrasts with the behavior observed in the reaction of $(\text{Me}_3\text{SiCH}_2)_2\text{GaCl}$ with $(\text{Me}_3\text{Si})_3\text{As}$, where both 1:1 and 2:1 mole ratios yield only mixed bridge compound **4**.

Crystals of **1** suitable for X-ray diffraction were obtained by recrystallization from a pentane solution. Crystallographic details are summarized in Table I. An ORTEP diagram showing the atom numbering scheme of **1** is presented in Figure 1. Table II gives the non-hydrogen atom fractional coordinates and equivalent isotropic thermal parameters, while Table IV gives selected bond lengths and angles.

In accord with its isomorphous gallium analogue, compound **1** contains a planar four-membered ring with slightly unequal bond lengths [$\text{In-As} = 2.733(2), 2.722(2) \text{ \AA}$]. The In-As-In angle is $94.57(5)^\circ$ and As-In-As angles are $85.21(4)$ and $85.65(4)^\circ$. The mean of the In-As bond lengths in **1** (2.728 \AA) is considerably greater than that at 2.676 \AA in $[\text{Me}_2\text{InAsMe}_2]_3$ (**5**),¹⁰ the only other compound containing In-As bonds to have its structure completely elucidated by single-crystal X-ray analysis.

Such bond length variation appears to be a general phenomenon among many group III-V dimers and trimers.¹¹ The planar nature of **1** and the pattern of exocyclic bond angles subtended at the arsenic and indium centers [$\text{Si-As-Si}' = 105.4(2)^\circ \ll \text{C-In-C}' = 125.8(5), 125.0(5)^\circ$] are similar to those of the gallium analogue, **3** [$\text{Si-As-Si}' = 103.66(6) \ll \text{C-Ga-C}' = 123.2(3), 124.6(3)^\circ$].

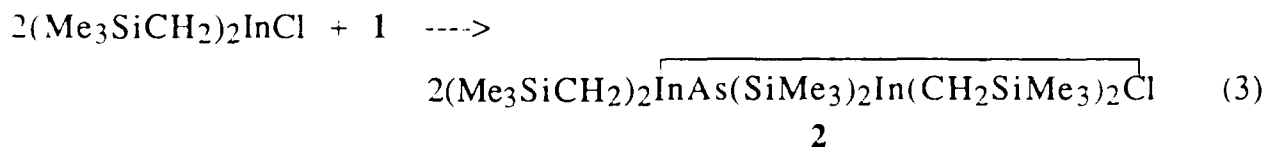
Cowley and Jones *et al.* have reported an indium-arsenic dimer of formula $[\text{Me}_2\text{InAsBu}^t_2]_2$ (**6**)¹², but no detailed crystallographic data have appeared. Both compounds **1** and **6** are colorless, crystalline materials which decompose at elevated temperatures (**1** decomposes to a black solid above 210°C , whereas, **6** is reported to melt with decomposition at 250 - 252°C).

Recrystallization of **2** from pentane provided crystals of **2** suitable for an X-ray structural study. Crystal data and data collection and refinement parameters are given in Table I. An ORTEP diagram of **2**, with the atom numbering scheme, is illustrated in Figure 2. Table III lists the non-hydrogen atom fractional coordinates and equivalent isotropic thermal parameters; Table V contains bond lengths and angles.

In **2**, the As and Cl atoms lie on a crystallographic C_2 symmetry axis and thus the $\overline{\text{In-As-In'-Cl}}$ ring is strictly planar. This ring geometry contrasts with that adopted in **4**, its gallium analogue, as well as in $\text{Ph}_2\overline{\text{GaAs}(\text{SiMe}_3)_2\text{GaPh}_2\text{Cl}}$ (**7**), both of which have the Cl atom displaced from the Ga-As-Ga' plane. The In-As bond distance at $2.677(1) \text{ \AA}$ in **2** is virtually identical to the mean In-As bond length at 2.676 \AA in trimer **5**¹⁰. The In-Cl-In' angle at $94.7(1)^\circ$ is larger than the Ga-Cl-Ga' bond angle in either **4** or **7** [$91.33(4)$ and $91.3(1)^\circ$, respectively]. Although **2** does not have the steric constraints associated with the additional trimethylsilyl

ligands . . 1, the exocyclic C-In-C' bond angle at 126.3(3)° in 2 is similar to the C-In-C' angles [125.8(5), 125.0(5)°] in 1.

It was observed by us that $[\text{Ph}_2\text{GaAs}(\text{SiMe}_3)_2]_2$ reacts cleanly with two mole equivalents of Ph_2GaCl to afford 7² and, more recently, we reported a similar conversion of 3 to its corresponding mixed-bridge species, 4 *via* reaction of 3 with two equivalents of $(\text{Me}_3\text{SiCH}_2)_2\text{GaCl}$ ³. Likewise, 1 reacts with two equivalents of $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ to afford 2 as the only product (eq 3). As evidenced by the ¹H NMR spectrum of the



reaction mixture, compound 1 reacts very quickly with $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$. Before all of compound 1 had dissolved completely, the only signals observed were those which resulted from $(\text{Me}_3\text{SiCH}_2)_2\text{InCl}$ and 2. This method of preparing 2 is preferable to dehalosilylation since the product is cleaner and can thus be recovered in greater yield.

Conclusions

The fact that 2 can be prepared readily by two routes provides additional evidence that mixed-bridge compounds may become a common species in the area of chemistry involving the heavier elements of Groups III and V. In addition, we believe it is significant that the specific methodology wherein a gallium-arsenic mixed-bridge compound results from reaction between a gallium-arsenic dimer and a dialkylgallium halide can be applied to the preparation of a like indium-arsenic compound from

analogous indium reactants. Further applications of this methodology, including efforts to prepare mixed-metal mixed-bridge compounds for use in subsequent studies, are being investigated in our laboratories.

Acknowledgment. The financial support for this work by the Office of Naval Research is gratefully acknowledged.

Supplementary Material Available: Tables of hydrogen atom coordinates and isotropic thermal parameters, anisotropic temperature factors, complete lists of interatomic distances and angles, including torsion angles, and equations of least-squares planes through groups of atoms for **1** and **2** (12 pages); a listing of observed and calculated structure amplitudes for **1** and **2** (35 pages). Ordering information is given on any current masthead page.

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Captions to Figures

Figure 1. ORTEP diagram of the solid-state structure of $[(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2]_2$ (1), with thermal ellipsoids at the 40% probability level; hydrogen atoms have been omitted for clarity.

Figure 2. ORTEP diagram of the solid-state structure of $(\text{Me}_3\text{SiCH}_2)_2\text{InAs}(\text{SiMe}_3)_2\text{In}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}$ (2), with thermal ellipsoids at the 40% probability level; hydrogen atoms have been omitted for clarity.

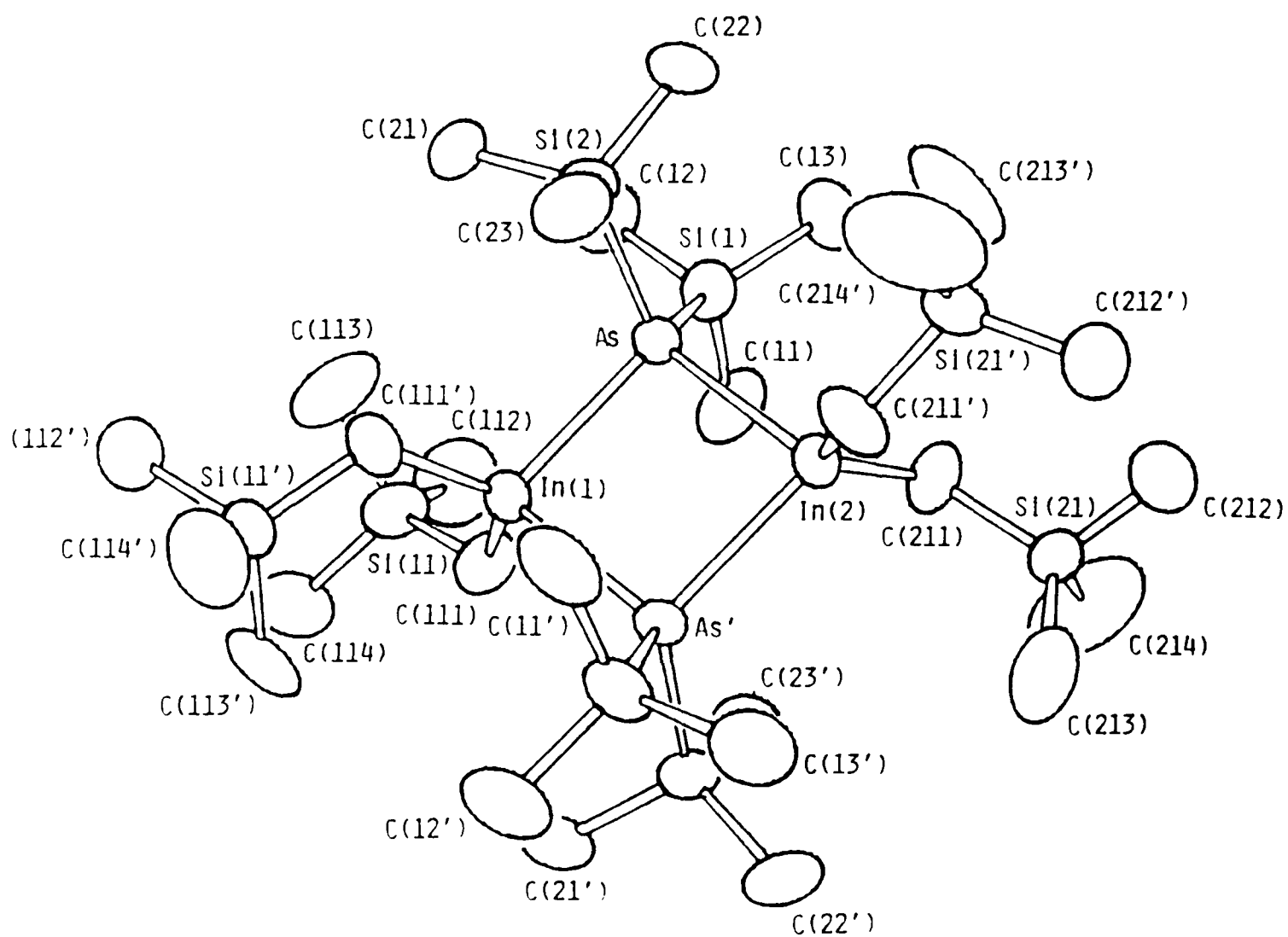


Figure 1

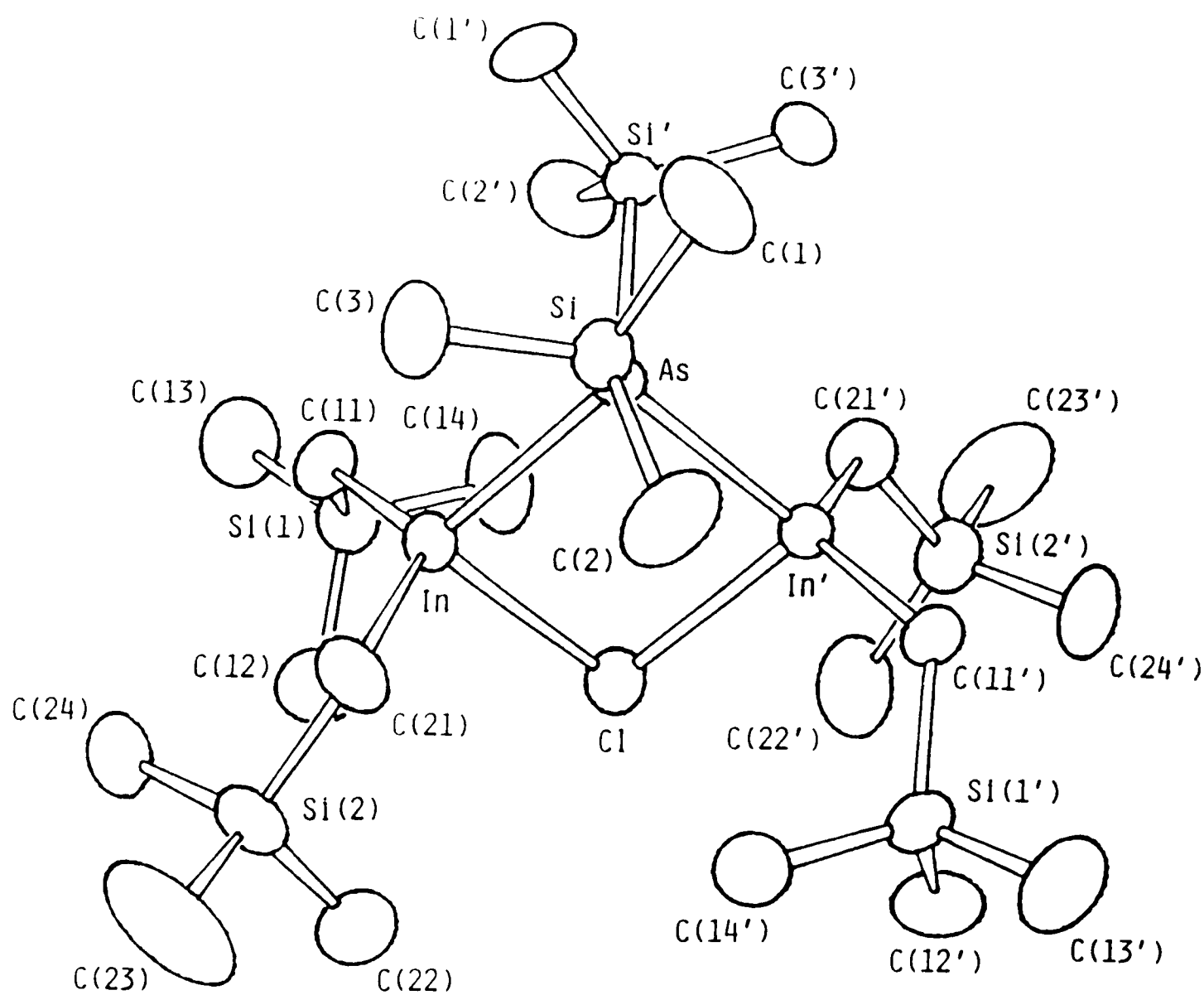
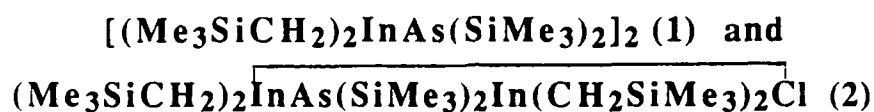


Figure 2

Table I. Crystallographic Data and Measurements^a for

	1	2
molecular formula	$\text{C}_{28}\text{H}_{80}\text{As}_2\text{In}_2\text{Si}_8$	$\text{C}_{22}\text{H}_{62}\text{AsClIn}_2\text{Si}_6$
formula weight	1021.12	835.27
crystal system	orthorhombic	monoclinic
space group	$Pbcn(D^{14}_{2h})$	$C2/c(C^6_{2h})$
a , Å	12.920(1)	19.233(3)
b , Å	21.618(2)	10.798(2)
c , Å	18.797(2)	20.819(3)
β , deg		105.91(1)
no. of orient. refls; θ , deg, range	25; 40-45	25; 40-46
V , Å ³	5250(1)	4158(2)
Z	4	4
D_{calcd} , g cm ⁻³	1.292	1.334
temp, °C	25	25
crystal dimensions, mm	0.20 x 0.20 x 0.26	0.30 x 0.40 x 0.70
$T_{\text{max}}:T_{\text{min}}$	1.00:0.70	1.00:0.25
radiation (wavelength)	Cu- $K\alpha$ (1.5418 Å)	Cu- $K\alpha$ (1.5418 Å)
μ , cm ⁻¹	105.1	123.4
scan type	ω -2 θ	ω -2 θ
scan width, deg	0.80 + 0.14tan θ	1.00 + 0.14tan θ
θ_{max} , deg	75	75

Table I. (continued)

intensity control rflns	441, 443, 443, 441	313, 531, 531, 313
variation; repeat time, h	-37%; 2	<2%; 2
no of rflns recorded	5386 (+h,+k,+l)	4388 (+h,+k, \pm l)
no of rflns retained, $I > 3.0\sigma(I)$	1877	3252
no of parameters refined	183	147
extinction correction	$1.5(1) \times 10^{-7}$	$2.1(1) \times 10^{-6}$
R, R_w^b	0.054, 0.071	0.067, 0.088
goodness-of-fit ^c	1.3	2.1
max shift; esd in final least-squares cycle	0.01	0.03
final $\Delta\rho(\text{e}/\text{\AA}^3)$ max; min	0.87; -1.57	2.5; -1.9

^aAn Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromator was used for all measurements. Crystallographic calculations were performed on PDP11/44 and MicroVAX II computers by use of the Enraf-Nonius Structure Determination Package (SDP).

^b $R = \sum ||F_o| - |F_c|| / \sum |F_o|$; $R_w = [\sum w (|F_o| - |F_c|)^2 / \sum w |F_o|^2]^{1/2}$; $\sum w \Delta^2 [w = 1/\sigma^2(|F_o|)$, $\Delta = (|F_o| - |F_c|)]$ was minimized.

^cGoodness-of-fit = $[\sum w \Delta^2 / (N_{\text{observations}} - N_{\text{parameters}})]^{1/2}$.

Table II. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for 1, with Estimated Standard Deviations in Parentheses.

atom	x	y	z	$B_{eq}(\text{\AA}^2)$
As	0.14199(10)	0.18627(6)	0.23718(7)	3.97(3)
In(1)	0.00000(-) ^a	0.27933(5)	0.25000(-) ^a	4.23(3)
In(2)	0.00000(-) ^a	0.09393(5)	0.25000(-) ^a	4.09(2)
Si(1)	0.2386(3)	0.1749(2)	0.1321(3)	5.5(1)
Si(2)	0.2644(3)	0.1908(2)	0.3292(3)	5.8(1)
Si(11)	0.0391(4)	0.3900(2)	0.1150(3)	5.3(1)
Si(21)	-0.0858(4)	-0.0211(2)	0.1307(3)	6.7(1)
C(11)	0.1501(14)	0.1895(8)	0.0567(9)	7.7(5)
C(12)	0.3506(13)	0.2304(8)	0.1292(11)	8.4(5)
C(13)	0.2933(12)	0.0938(7)	0.1272(10)	7.3(5)
C(21)	0.3332(13)	0.2661(8)	0.3283(11)	9.0(6)
C(22)	0.3595(14)	0.1262(8)	0.3202(10)	8.6(5)
C(23)	0.1939(14)	0.1823(8)	0.4163(9)	7.9(5)
C(111)	-0.0366(13)	0.3249(6)	0.1507(10)	6.9(4)
C(112)	0.0324(20)	0.3903(13)	0.0185(12)	16(1)
C(113)	0.1780(14)	0.3866(8)	0.1405(14)	12.8(7)
C(114)	-0.0123(19)	0.4614(8)	0.1433(21)	20(1)
C(211)	-0.0036(12)	0.0468(6)	0.1459(8)	5.9(4)

Table II. (continued)

C(212)	-0.0039(15)	-0.0910(8)	0.1122(17)	12.7(8)
C(213)	-0.1751(15)	-0.0366(8)	0.2085(12)	10.6(6)
C(214)	-0.1683(18)	-0.0101(10)	0.0496(11)	11.5(7)

^a Fixed by symmetry

Table III. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for 2, with Estimated Standard Deviations in Parentheses.

atom	x	y	z	$B_{eq}(\text{\AA}^2)$
As	0.00000(-) ^a	0.0313(1)	0.25000(-) ^a	2.77(2)
In	0.09994(2)	0.20343(5)	0.24914(2)	3.12(1)
Cl	0.0000(-) ^a	0.3677(3)	0.2500(-) ^a	4.69(6)
Si	0.0419(1)	-0.0958(2)	0.3447(1)	3.58(4)
Si(1)	0.1009(1)	0.3459(3)	0.0973(1)	4.45(5)
Si(2)	0.2302(1)	0.3679(3)	0.3624(1)	5.27(6)
C(1)	-0.0122(6)	-0.2397(10)	0.3378(6)	7.0(3)
C(2)	0.0352(6)	-0.0065(13)	0.4184(5)	7.0(3)
C(3)	0.1373(5)	-0.1350(10)	0.3532(5)	5.6(2)
C(11)	0.1294(4)	0.2160(8)	0.1563(4)	4.1(2)
C(12)	0.1315(7)	0.4968(12)	0.1382(6)	7.0(3)
C(13)	0.1429(7)	0.3273(15)	0.0270(5)	7.9(3)
C(14)	0.0018(6)	0.3467(13)	0.0638(6)	7.4(3)
C(21)	0.1716(5)	0.2281(10)	0.3485(3)	4.8(2)
C(22)	0.1743(7)	0.5104(13)	0.3513(8)	9.0(4)
C(23)	0.2954(9)	0.3655(20)	0.4465(7)	13.3(5)
C(24)	0.2833(6)	0.3674(13)	0.3005(7)	7.5(3)

^a Fixed by symmetry.

Table IV. Selected Bond Distances (Å) and Angles (deg) for 1, with Estimated Standard Deviations in Parentheses.

Bond Lengths			
As-In(1)	2.733(2)	In(1)-C(111)	2.16(2)
As-In(2)	2.722(2)	In(2)-C(211)	2.21(1)
As-Si(1)	2.349(5)		
As-Si(2)	2.346(5)		

Bond Angles			
In(1)-As-In(2)	94.57(5)	As-In(1)-As'	85.21(4)
In(1)-As-Si(1)	120.5(1)	As-In(1)-C(111')	105.3(4)
In(1)-As-Si(2)	110.9(1)	C(111)-In(1)-C(111')	125.8(5)
In(2)-As-Si(1)	110.8(2)	As-In(2)-C(211)	105.9(4)
In(2)-As-Si(2)	114.8(1)	As-In(2)-As'	85.65(4)
Si(1)-As-Si(2)	105.4(2)	As-In(2)-C(211')	113.8(4)
As-In(1)-C(111)	113.9(4)	C(211)-In(2)-C(211')	125.0(5)

Table V. Selected Bond Distances (\AA) and Angles (deg) for 2, with Estimated Standard Deviations in Parentheses.

Bond Lengths			
As-In	2.677(1)	In-C(11)	2.162(9)
As-Si	2.356(2)	In-C(21)	2.165(6)
In-Cl	2.619(2)		
Bond Angles			
In-As-In'	92.07(4)	Cl-In-C(21)	100.7(3)
In-As-Si	109.18(5)	C(11)-In-C(21)	126.3(3)
In-As-Si'	118.71(6)	In-Cl-In'	94.7(1)
Si-As-Si'	108.73(8)	As-Si-C(1)	111.6(3)
As-In-Cl	86.59(6)	As-Si-C(2)	107.8(4)
As-In-C(11)	114.5(2)	As-Si-C(3)	108.5(3)
As-In-C(21)	110.4(3)	In-C(11)-Si(1)	122.7(4)
Cl-In-C(11)	109.6(2)	In-C(21)-Si(2)	116.7(4)

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